# IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN THE APPLICATION OF: MATTHEW R. SIVIK ET AL.

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TITLE: FUNCTIONALISED POLYMER COMPOSITION FOR GREASE

Wickliffe, Ohio

Dated: December 30, 2008

### Declaration Under Rule 1.132

Sir,

I Matthew R. Sivik a named inventor of the above-identified application hereby declare:

I, Matthew R. Sivik, have been employed by The Lubrizol Corporation as a chemist since 1991. I obtained a PhD. from The Ohio State University in 1991 in the field of organic chemistry and have 17 years experience in research in the preparation and formulation of additives and polymers for use in lubricants and greases.

In order to demonstrate the advantages of the grease compositions of the present invention, the following comparative study was undertaken under my supervision:

## Grease Composition of the Invention (GR1)

Calcium sulfonate grease is prepared in a similar manner to example 1 of the patent application for this invention. The calcium sulphonate used in this example has a 400 total base number and contains 42% diluent oil. The calcium sulfonate grease differs only in the amount of calcium sulphonate present (66.36 wt %), and that the

grease contains 2.22 wt % of dodecyl succinic acid. The amount of dodecyl succinic acid delivers 11 milli moles of succinic groups per 100 g of the total amount of calcium sulfonate and dodecyl succinic acid. This sample contains a hydrocarbyl substituted dicarbonyl derivate, as specified in the claims, specifically dodecyl succinic acid.

#### Comparative Grease Composition 1 (CGC1)

Calcium sulfonate grease is prepared in a similar manner to example 1 of the patent application for this invention. The calcium sulphonate used in this example has a 400 total base number and contains 42% diluent oil. The calcium sulfonate grease differs only in the amount of calcium sulfonate present (63.5 wt %), and that the grease contains 0.68 wt % of succinic anhydride. The succinic anhydride is hydrolyzed in situ to succinic acid. The amount of succinic anhydride delivers 11 milli moles of succinic groups per 100 g of calcium sulphonate and succinic anhydride. This sample contains a non-substituted dicarbonyl derivate, specifically succinic anhydride.

## Comparative Grease Composition 2 (CGC2)

Calcium sulfonate grease is prepared in a similar manner to example 1 of the patent application for this invention. The calcium sulphonate used in this example has a 400 total base number and contains 42% diluent oil. The calcium sulphonate grease differs only in the amount of calcium sulphonate present (63 wt %), and that the grease contains 1.36 wt % of succinic anhydride. The succinic anhydride is hydrolyzed in situ to succinic acid. The amount of succinic anhydride delivers 20 milli moles of succinic groups per 100 g of calcium sulphonate and succinic anhydride. This sample contains a non-substituted dicarbonyl derivate, specifically succinic anhydride.

#### Grease Evaluation

The grease compositions described above were evaluated by the standard ASTM method D4049. The test measures the resistance of grease to water spray. A pre-weighed stainless steel panel is evenly coated with about 8mm of grease. The panel is then reweighed. The coated stainless steel panel is then placed in a water spray for about 5 minutes. The water is preheated to about 38 °C and held at constant

temperature. The water pressure pump is held at about 276 kPa (equivalent to about 40 psi). The panel is removed from the spray and heated in an oven for about 1 hour at about 66 °C. The panel is then removed from the oven, allowed to cool and is reweighed in order to determine the amount of grease removed from the sample during the testing. The results obtained for the grease compositions are shown in the Table below.

ASTM D4049 Results

Example	% Grease Removed by Water Spray
GR1	14.1
CGC1	26.4
CGC2	22.0

The results indicate the calcium sulfonate grease of the present invention, which contains a hydrocarbyl substituted dicarbonyl derivate, has a reduced water spray-off compared with either comparative example, which do not contain hydrocarbyl substituted dicarbonyl derivates. This means that the calcium sulphonate grease of the present invention is better able to remain on a surface, compared with calcium sulfonate greases not encompassed by the present claims.

I further declare that all statements herein made of my own knowledge are true and all statements herein made on information and belief are believed to be true. I understand that wilful false statements and the like are punishable by fine or imprisonment or both (18 U.S.C. 1001) and may jeopardize the validity of the application or any patent issuing thereon.

Matthew R. Sivik